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METHOD OF TEST FOR RECOVERING ASPHALTIC MATERIALS BY A MODIFIED (IA) ABSON RECOVERY PROCEDURE

CAUTION: Prior to handling test materials, performing equipment setups, and/or conducting this method, testers are required to read "**SAFETY AND HEALTH**" in Section H of this method. It is the responsibility of the user of this method to consult and use departmental safety and health practices and determine the applicability of regulatory limitations before any testing is performed.

A. SCOPE

This method covers the recovery of bitumen from asphaltic mixtures with trichloroethylene by the Industrial Asphalt Modified Abson Recovery Method. The bitumen is recovered with substantially the same properties as possessed in the asphaltic mixture.

B. APPARATUS

1. Centrifuge: Batch unit capable of exerting a minimum centrifugal force of 770 times gravity or continuous unit capable of exerting a minimum centrifugal force of 3,000 times gravity.

2. Centrifuge Tubes: Appropriate wide-mouth bottles to fit centrifuge unit used.

3. Distillation Apparatus: As shown in Figure 1 and consisting of the following:

   a. Distilling Flask, 2000 mL, three necks with Ⓢ 24/40 joints modified to angle type with centerline of side necks angled to center of bottom of flask. Modified flasks are available from: SCI-TECH Glassblowing Company, 8930 Eton Avenue, Canoga Park, CA 91304.

   b. Connecting tube, 3-way Ⓢ 24/40 joints, except thermometer opening is Ⓢ 10/30 joint.

   c. Condenser, with Ⓢ 24/40 joints, jacket length 200 mm minimum.

   d. Adapter, with Ⓢ 24/40 joint.

   e. Thermometer, 76.2 mm immersion, with Ⓢ 10/30 joint, range – 10 to 45°C.

   f. Thermometer, ASTM Designation: 45°C, softening point.

   g. Aerator tube, fabricated from 6.4 mm OD stainless steel tubing, with three No. 60 drill size holes drilled through both sides of the tubing at distances of 6.4 ± 1 mm, 31.8 ± 1 mm and 57.2 ± 1 mm from sealed tip. The aerated end of the tubing shall be bent to conform to the bottom of the distillation flask for a length of 76 ± 5 mm.

   h. Thermometer supports (2) with Ⓢ 24/40 joint.

   i. Electric heating mantle, to fit the 2000 mL flask, with a variable temperature control.

   j. Flow meter and valve, for accurately measuring quantity of CO_2 gas up to 1000 ± 50 mL/min.

   k. Distillate receiver – 2000 mL graduated beaker or flask.

   l. Mechanical convection oven capable of heating to 150°C.

C. REAGENTS AND MATERIALS

1. Carbon dioxide gas in a pressurized tank with a pressure-reducing regulator, or other convenient source.

2. Trichloroethylene, reagent grade or double distilled technical grade, Type I, Federal Specification O-T-634.

3. Aluminum foil, heavy-duty.

D. SAMPLE

The sample shall consist of the solution from a previous extraction of an asphaltic mixture, using trichloroethylene as the solvent. For a satisfactory extraction method, refer to ASTM Designation: D-2172 (Method A, B, or C) or other suitable method. The asphaltic mixture sample must be of sufficient quantity to yield between 125 to 250 g of recovered bitumen. The extraction of asphalt using California Test 310 or another suitable method will provide the information needed to establish the sample size, although 3500 g is usually sufficient.

Sample identification is essential. Make sure that identification is maintained throughout the process.

E. PROCEDURE

1. If a batch centrifuge is used, centrifuge the solution from the previous extraction for a minimum of 30 min at 770 times gravity. If a continuous centrifuge is used, the extract solution shall be charged at a rate not to exceed 150 mL/min, while the unit is operating at a speed calculated to produce a centrifugal force of not less than 3000 times gravity. When using either centrifuge method, the solution must be relatively dilute (e.g., approximately 10 parts trichloroethylene to 1 part bitumen) (Note 1).

2. Obtain the tare mass for the distillation flask, aeration tube, thermometer, and both supports (shown assembled in Figure 1).

3. Pour the centrifuged solution into the distillation flask. Rinse the centrifuge container with trichloroethylene to transfer all of the asphalt. No more than 1700 mL of solution should be added to the flask (Note 2).

4. Start the CO_2 flow rate at 100 mL/min, wrap that portion of distillation flask above the heating mantle with a double thickness of heavy-duty aluminum foil up to the bottom of the outlet necks, and turn the heating temperature control to a predetermined setting to obtain a slow, even boil (see notes on Figure 1). Distill until the major portion of the trichloroethylene solvent has been removed (Note 3).

   a. Method A: When the residue temperature reaches 127°C, increase the CO_2 flow rate to 1000 mL/min and lower the voltage to a predetermined setting to bring the residue temperature to 150°C. As the residue temperature approaches 150°C, monitor the adapter column for condensation of light oils from the bitumen. Maintain the residue temperature at 150 ± 8°C for 30 min with the CO_2 flow rate at 1000 mL/min.

   b. Method B: Raise the residue temperature slowly to 163°C while continually monitoring the adapter column for oil condensation. If no condensation is observed, maintain this 163°C for 30 min with the CO_2 flow rate at 1000 mL/min.

5. At the end of 30 min, shut off the heat and CO_2, dismantle the distillation apparatus and remove the adapter column from the center neck of the distillation flask. Place the flask assembly, including the aeration tube, thermometer, and supports, upright in a 150°C oven for 20 min to allow all of the entrained CO_2 to escape (Note 4). Sometime after the CO_2 has been turned off, roll the asphalt in the flask for two or three revolutions. This provides a larger surface area that allows the solvent to escape easier. Weigh the flask assembly (± 0.1 g) and return it to 150°C oven. Check the mass at 10 min intervals until a constant mass is reached.

6. Weigh the flask assembly as described above to determine the final mass. Calculate the bitumen content of the mixture and perform appropriate tests on the residue.

7. When preparing the recovered asphalt for testing, heat the residue on a hot plate (190 ± 10°C) to:

   a. Remove any stearic hardening that may have occurred.

b. Release any solvent that may remain trapped in the residue.

Stir the residue with a thermometer, maintaining a temperature between 150 and 160°C until the frequency of the bubble development reduces to 4 or 5 per second. This process is significant when the residue is very hard; e.g., having a 25°C penetration less than 15.

F. NOTES

1. Since ash contents greater than 1 % greatly affect the accuracy of tests performed on the residue, it is desirable in all recoveries, and mandatory in referee or research work, to conduct ash content determinations on the recovered residue. The centrifuging method is suspect if the ash content is greater than 1 %. If this is encountered, a repeat recovery procedure should be performed with special attention to the centrifuging method.

2. If the extraction method requires more than 1700 mL of solvent, a separatory funnel may be used in the thermometer opening of the distillation flask to add the remaining solution during primary distillation. The separatory funnel stopcock should be adjusted to maintain a constant level in the distillation flask. *DO NOT BOIL LEVEL DOWN AND ADD COLD SOLUTION TO HOT RESIDUE.* As soon as all of the solution is added to the distillation flask, replace separatory funnel with the thermometer and support, and proceed.

3. Since all extracted bitumen can, and does, vary due to age, mixing temperature and source, the distillation temperature must be governed by the initial boiling point of the material involved. This is the thought behind using a distillation trap and the thermometer to indicate distillate vapor temperature. *If oil condensation is observed in either of the above routines, the residue temperature MUST be reduced until vapor other than solvent vapor is no longer indicated.*

   When the viscosity of the recovered bitumen at 60°C is known or suspected of being below 1000 Pa•s, use Method A. If it is expected to be above 1000 Pa•s, use Method B.

4. If no oven is immediately available at the end of the 30 min, shut off the CO_2, partially dismantle the distillation apparatus by removing the adapter column from the center neck of the distillation flask, and maintain the final distillation temperature in the flask for an additional 20 to 30 min to allow the escape of all entrained CO_2.

G. REPORTING

Record mass and temperature data during the recovery process. Report this information and results of tests performed upon the residue on Form TL-394.

H. SAFETY AND HEALTH

The distillation routine must be performed in a well-ventilated area or under an appropriate fume hood in order to comply with the trichloroethylene vapor exposure guidelines.

Care should be exercised that exposure of skin to trichloroethylene be limited by the wearing of protective gloves and/or clothing.

Prior to handling, testing or disposing of any waste materials, testers are required to read: Part A (Section 5.0), Part B (Sections: 5.0, 6.0 and 10.0) and Part C (Section 1.0) of Caltrans Laboratory Safety Manual. Users of this method do so at their own risk.

REFERENCES:
California Test 310
ASTM Standards D-2172, Test for Quantitative Extraction of Bitumen from Bituminous Paving Mixtures.
E-1, Specification for ASTM Thermometers.

End of Text (California Test 380 contains 4 Pages)

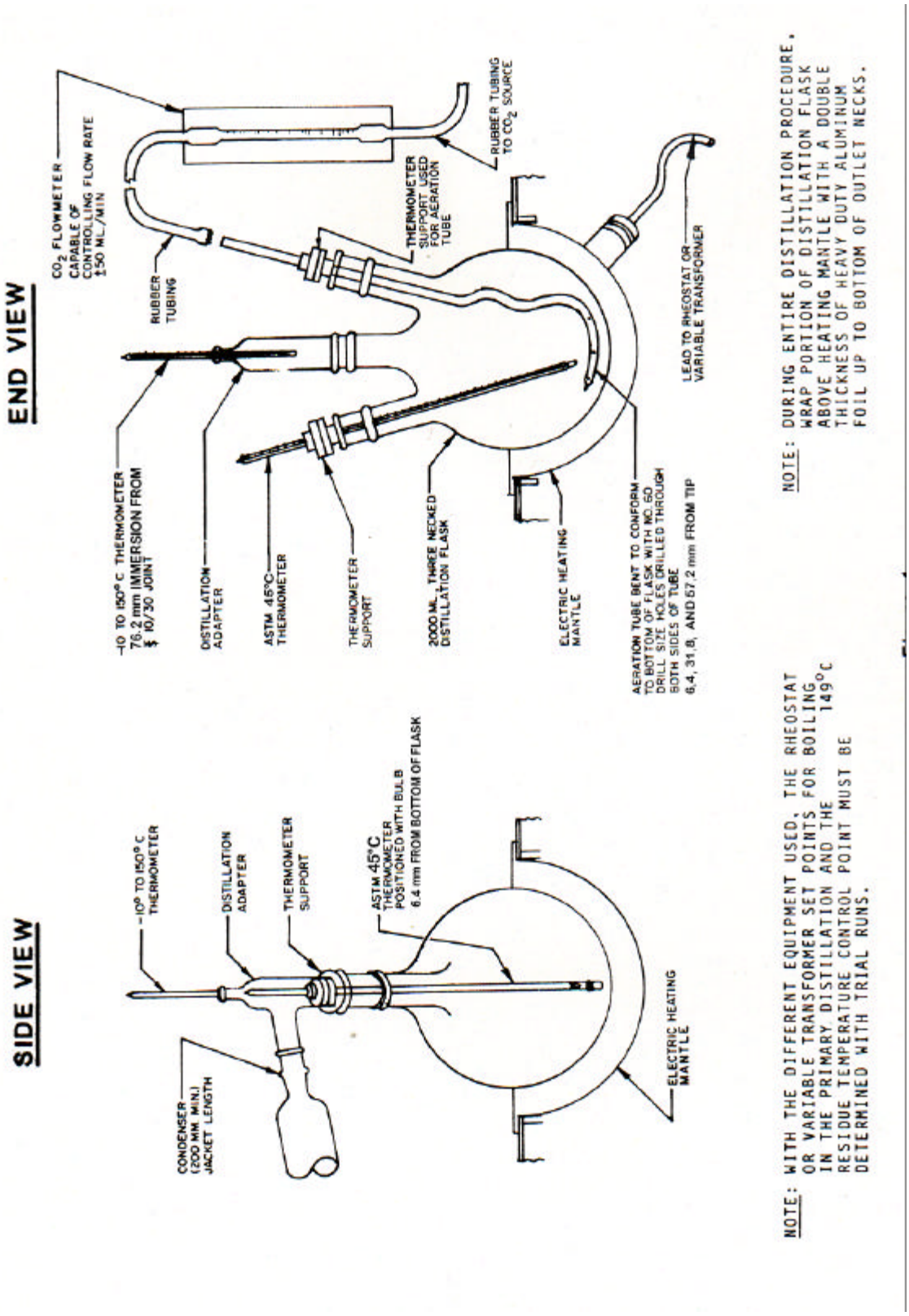
END VIEW
CO_2 FLOWMETER CAPABLE OF CONTROLLING FLOW RATE ±50 ML/MIN
RUBBER TUBING
RUBBER TUBING TO CO_2 SOURCE
THERMOMETER SUPPORT USED FOR AERATION TUBE
-10 TO 150° C THERMOMETER 76.2 mm IMMERSION FROM ∮ 10/30 JOINT
DISTILLATION ADAPTER
ASTM 45°C THERMOMETER
THERMOMETER SUPPORT
2000 ML, THREE NECKED DISTILLATION FLASK
ELECTRIC HEATING MANTLE
AERATION TUBE BENT TO CONFORM TO BOTTOM OF FLASK WITH NO. 50 DRILL SIZE HOLES DRILLED THROUGH BOTH SIDES OF TUBE 6.4, 31.8, AND 67.2 mm FROM TIP
LEAD TO RHEOSTAT OR VARIABLE TRANSFORMER
NOTE: DURING ENTIRE DISTILLATION PROCEDURE, WRAP PORTION OF DISTILLATION FLASK ABOVE HEATING MANTLE WITH A DOUBLE THICKNESS OF HEAVY DUTY ALUMINUM FOIL UP TO BOTTOM OF OUTLET NECKS.
SIDE VIEW
-10° TO 150° C THERMOMETER
DISTILLATION ADAPTER
THERMOMETER SUPPORT
ASTM 45°C THERMOMETER POSITIONED WITH BULB 6.4 mm FROM BOTTOM OF FLASK
CONDENSER (200 MM MIN.) JACKET LENGTH
ELECTRIC HEATING MANTLE
NOTE: WITH THE DIFFERENT EQUIPMENT USED, THE RHEOSTAT OR VARIABLE TRANSFORMER SET POINTS FOR BOILING IN THE PRIMARY DISTILLATION AND THE RESIDUE TEMPERATURE CONTROL POINT MUST BE DETERMINED WITH TRIAL RUNS. 149°C

FIGURE 1